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JULY 1963

CLEAVAGE SURFACE ENERGY OF {100} MAGNESIUM OXIDE

By
A. R. C. WESTWOOD
and
D. L. GOLDHEIM

SECOND TECHNICAL REPORT
OFFICE OF NAVAL RESEARCH PROJECT
NONR-4162(00)

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Second Technical Report to O.N.R.

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A. R. C. Westwood and D. L. Goldheim

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ABSTRACT

The modification of the Gilman cleavage technique described recently by Westwood and Hitch has been used to determine the surface energy, γ_o, of {100} MgO at 298°K. This was found to be 1150 $\pm$ 80 ergs/cm^2, in good agreement with previous experimental estimates of γ_o, but somewhat lower than theoretical estimates of this quantity. Metallographic studies were also made of the plastic relaxation which occurs in the vicinity of the crack tip when cleavage cracks are repropagated, and a correlation between the extent of plastic relaxation, crack length and the measured cleavage surface energy was observed.

I. INTRODUCTION

The modification of the Gilman[1] cleavage technique described recently by Westwood and Hitch[2], provides a convenient method of determining the surface energy of the cleavage planes of materials which possess some ductility at the temperature of testing, for example, certain ionic crystals at room temperature.

Briefly, the technique involves determining the load P to propagate previously initiated cracks of length L_o in specimens of the form and dimensions illustrated in Fig. 1. The true surface energy, γ_o, is then derived from Eq. (1)[2]

$$\left(\frac{1}{6P^2L_o^2/Ew^2t^3}\right) = \frac{1}{\gamma_A} = \frac{1}{\gamma_o} + \frac{\alpha E}{4\gamma_o G}\left(\frac{t}{L_o}\right)^2 \qquad (1)$$

by plotting values of the reciprocal of the "apparent" surface energy, $(1/\gamma_A)$, versus $(t/L_o)^2$. Such a plot should be linear, and of intercept $(1/\gamma_o)$ when extrapolated to $(t/L_o)^2 = 0$. E and G are respectively the Young's modulus and shear modulus of the material, and α is an experimental coefficient [3].

However, in previous investigations[1,2,4] utilizing the cleavage technique, it has been observed that if specimens are used which contain

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[1]J. J. Gilman, J. Appl. Phys., 31, 2208 (1960).

[2]A. R. C. Westwood and T. T. Hitch, J. Appl. Phys., 34 (1963) in press.

[3]S. Timoshenko, Theory of Elasticity (McGraw Hill, New York, 1934), p. 33.

[4]A. R. C. Westwood and M. H. Kamdar, Phil. Mag., 8, 787 (1963).

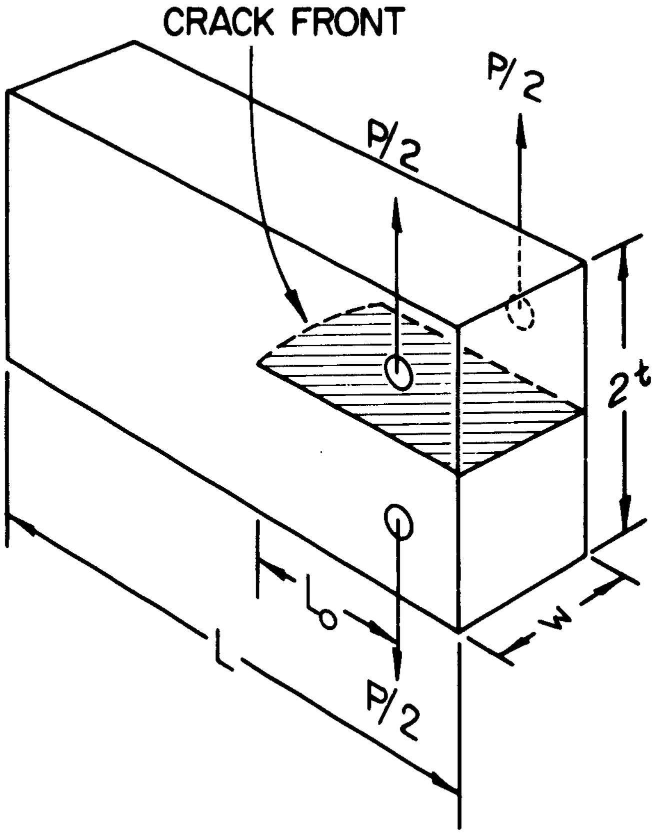

Figure 1. Schematic of specimens used to determine γ_0 .

cracks of length greater than 2-3t, then the values of γ_o determined are often several (0-10) times larger than either theoretical estimates of γ_o or values determined by other experimental methods. It has been suggested[2] that this is because the propagation of "long" cracks in such materials as LiF[1], KCl[2], Zn[1,4] and MgO[1] at room temperature is preceded by plastic relaxation at the crack tip, and plastically blunted cracks require a greater stress to induce repropagation.

To investigate this possibility, a metallographic study has been made of the plastic deformation which accompanies the repropagation of cleavage cracks in MgO. In other experiments, the cleavage technique outlined above has been used to determine γ_o {100} MgO at room temperature.

II. EXPERIMENTAL PROCEDURE

1. Determination of γ_o {100} MgO.

Monocrystal specimens of MgO were cleaved from large polycrystalline blocks obtained from the Norton Company. Though these crystals were colorless, their purity was probably not greater than 99.9%. Specimen dimensions, Fig. 1, were typically: L = 20-30mm; 2t = 6-20mm; and w = 2-4mm. Cleavage cracks were initiated at room temperature by means of a hand-held chisel, and were prevented from propagating completely through the specimens by the application of a small compressive stress perpendicular to the direction of propagation. Each specimen was then promptly mounted in a crack-propagation jig[2] attached to the cross-head of an Instron machine, taking care to ensure that the cleavage plane was accurately perpendicular to the loading axis. The crack length, L_o, was set at an appropriate value, ranging from 2-15mm, so as to provide a pre-determined value of $(t/L_o)^2$ between 0.1 and 16.0. The Instron crosshead speed was 0.005"/min., producing a rate of loading of ∿20 gm/sec. The load P to repropagate the crack, indicated by a sharp drop in the load-deflection curve, was noted. After fracture, an accurate value of L_o was obtained by microscopic examination of the fracture surfaces, and the values of w and t were also obtained optically. Finally, the factors $(1/\gamma_A)$ and $(t/L_o)^2$ were computed. The value of Young's modulus used (E <100> MgO = 24.84×10^{-11} dynes/cm^2) was that of Durand[5] and Chung[6].

2. Metallographic studies

Various experiments were performed to investigate the distribution of dislocations in the vicinity of the crack tip before and after propagation of the crack, and to correlate any observed variation in distribution with such factors as crack length, the ratio $(t/L_o)^2$, and the corresponding value of γ_A. The etchant used was that developed by Stokes et al[7] and consisted of 5 parts of saturated ammonium chloride, one part of sulphuric acid and one part of distilled water. Specimens were usually etched for 5-10 minutes at room temperature.

Particular experimental procedures will be described in III.2.

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[5] M. A. Durand, Phys. Rev., 50, 449 (1936).

[6] D. H. Chung, Phil. Mag., 8, 833 (1963).

[7] R. J. Stokes, T. L. Johnston and C. H. Li, Phil. Mag., 3, 718 (1958).

III. RESULTS AND DISCUSSION

1. Determination of γ_o {100} MgO.

The experimental data is presented in Fig. 2. In accord with Eq. (1) the variation of $(1/\gamma_A)$ with $(t/L_o)^2$ was found to be linear. Data was satisfactorily reproducible when $(t/L_o)^2$ lay between 0.4 and 3.0, but scatter increased appreciably when $(t/L_o)^2$ was greater than ~4.0. This increase in scatter with $(t/L_o)^2$ is associated with the increased importance of errors in the measurement of L_o when L_o is small (since cracks fronts are always somewhat rounded), with the plotting of the reciprocal of γ_A as ordinate, and with the increasing inapplicability of the theory used to derive Eq. (1) as L_o decreases with respect to t.[2] The line drawn in Fig. 2 is considered to be a best fit to the experimental points, and this intercepts the ordinate axis, Fig. 2(b), at 8.7×10^{-4} cm^2/erg, with a possible error of $\pm$ 0.6×10^{-4} cm^2/erg. Thus the surface energy, γ_o , for the {100} faces of MgO at 298°K is found to be 1150 $\pm$ 80 ergs/cm^2.

This value is in reasonable agreement with previous experimental determinations of γ_o , Table I, and is considered to be a more reliable estimate of γ_o than previously available. However, as noted and discussed previously[2], values of the true surface energy determined by the cleavage technique are usually somewhat lower than the theoretical estimates of γ_o for unpolarized* surfaces, Table I.

*Since the surface polarization and relaxation phenomenon[13,14] occurs after the crack has propagated, the decrease in surface energy associated with this process will not be recorded by a cleavage technique[2].

[13] J. E. Lennard-Jones and B. M. Dent, Proc. Roy. Soc. (London) A121, 247 (1928).

[14] B. M. Dent, Phil. Mag., 8, 530 (1929).

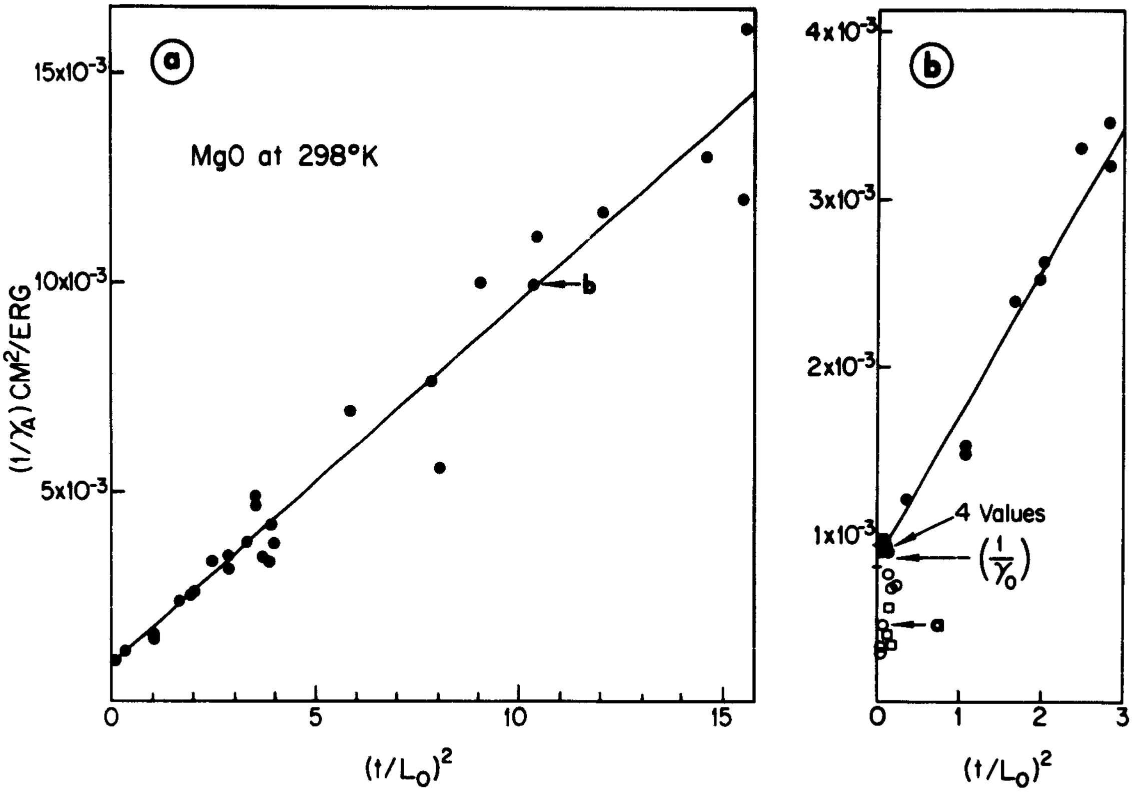

Figure 2. Effect of variations in crack length L_o and specimen dimension t on the apparent surface energy γ_A of (100) MgO at 298°K. The letters (a) and (b) refer to specimens illustrated in Figs. 5(a) and 5(b) respectively.

TABLE I - Summary of Data on Surface Energy of (100) MgO.

Author	Method	Value (ergs/cm^2)
Theoretical Estimates		
Born & Stern[8]	Elementary electrostatic approach	1440(0°K)*
Lennard-Jones & Taylor[9]	As above	1362(0°K)
Glauberman[10]	As above	1450(0°K)† 1230(0°K)† ‡
Experimental Determinations		
Jura & Garland[11]	Heat of Solution of MgO powder	1000(298°K) 1040(0°K)
Gilman[1]	Cleavage Technique. Value is author's choice on basis of limited number of tests.	1200(77°K)
Present work	Cleavage Technique with Data Extrapolation to determine γ_o.	1150±80(298°K)

* Quoted in ref. 9.

† Calculated from Glauberman's theory using ionic radii from Wyckoff[12]

‡ Value for polarized surface.

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[8] M. Born and O. Stern, Sitzb. Preuss. Akad. Wiss., 48, 901 (1919).

[9] J. E. Lennard-Jones and P. A. Taylor, Proc. Roy. Soc. (London), A109, 476 (1925).

[10] A. E. Glauberman, Zhur. Fiz. Khim., 23, 124 (1949); Nat. Res. Council of Canada, Tech. Transl. TT-111 (1949).

[11] G. Jura and C. W. Garland, J. Am. Chem. Soc., 74, 6033 (1952).

[12] R. W. G. Wyckoff, Crystal Structures (Interscience 1948).

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[11] G. Jura and C. W. Garland, J. Am. Chem. Soc., 74, 6033 (1952).

[12] R. W. G. Wyckoff, Crystal Structures (Interscience 1948).

The data represented by open squares (after Gilman[1]) and open circles (present work) at $(t/L_o)^2 \leq 0.2$ illustrate the manner in which tests performed with relatively long cracks lead to inconsistent and often anomalously high values of γ_A, and hence, if one is unaware of this phenomenon, to possibly inaccurate estimates of γ_o. Values of γ_A for cracks of $(t/L_o)^2 < 0.2$ ranged from 1100 ergs/cm^2 to 3300 ergs/cm^2.

2. Plastic Relaxation at the Crack Tip

MgO crystals were partially cleaved in the manner described in the preceding section and then recleaved, as illustrated in Fig. 3, into two halves I and II.

Crystals I were etched to reveal the distribution of dislocations at and around the tip of the stationary crack. Crystals II were mounted in the testing machine, the crack length adjusted to an appropriate value of L_o (either $L_o < t$ or $L_o \approx 3t$) and the crack repropagated. Both halves of crystals II were then etched and examined.

Figure 4 compares the dislocation distribution on matching faces A and B (Fig. 3) for a specimen of $L_o \approx 3t$. In Fig. 4(a) (crystal I, Face B) the tip of the unpropagated crack, as revealed by the discontinuity in the cleavage steps, is at Y. However, the projection of crack XY is revealed as the row of etch pits, YZ. These may be associated with {100} <100> type sessile dislocations introduced during crack healing, as discussed by Shaskolskaya et al[15]. Fig. 4(b), (crystal II, Face A) illustrates a phenomenon termed a dislocation "wing",* and found to be characteristically associated

*Dislocation wings have also been observed in KCl, LiF and NaCl.

[15] M. P. Shaskolskaya, W. Yen-Wen and K. Shu-chao, Soviet Physics (Crystallography), 6, 483 (1962).

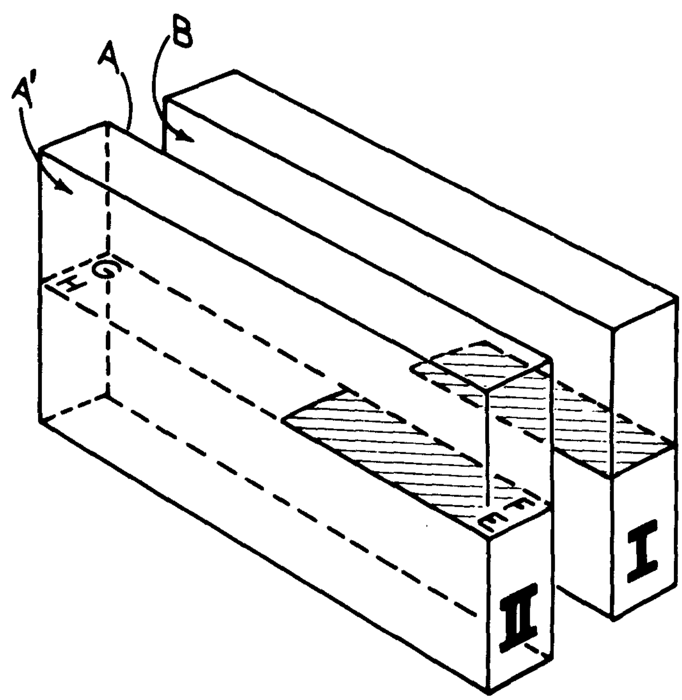

Figure 3. Illustrating manner in which partially cleaved specimens were further cleaved for metallographic study.

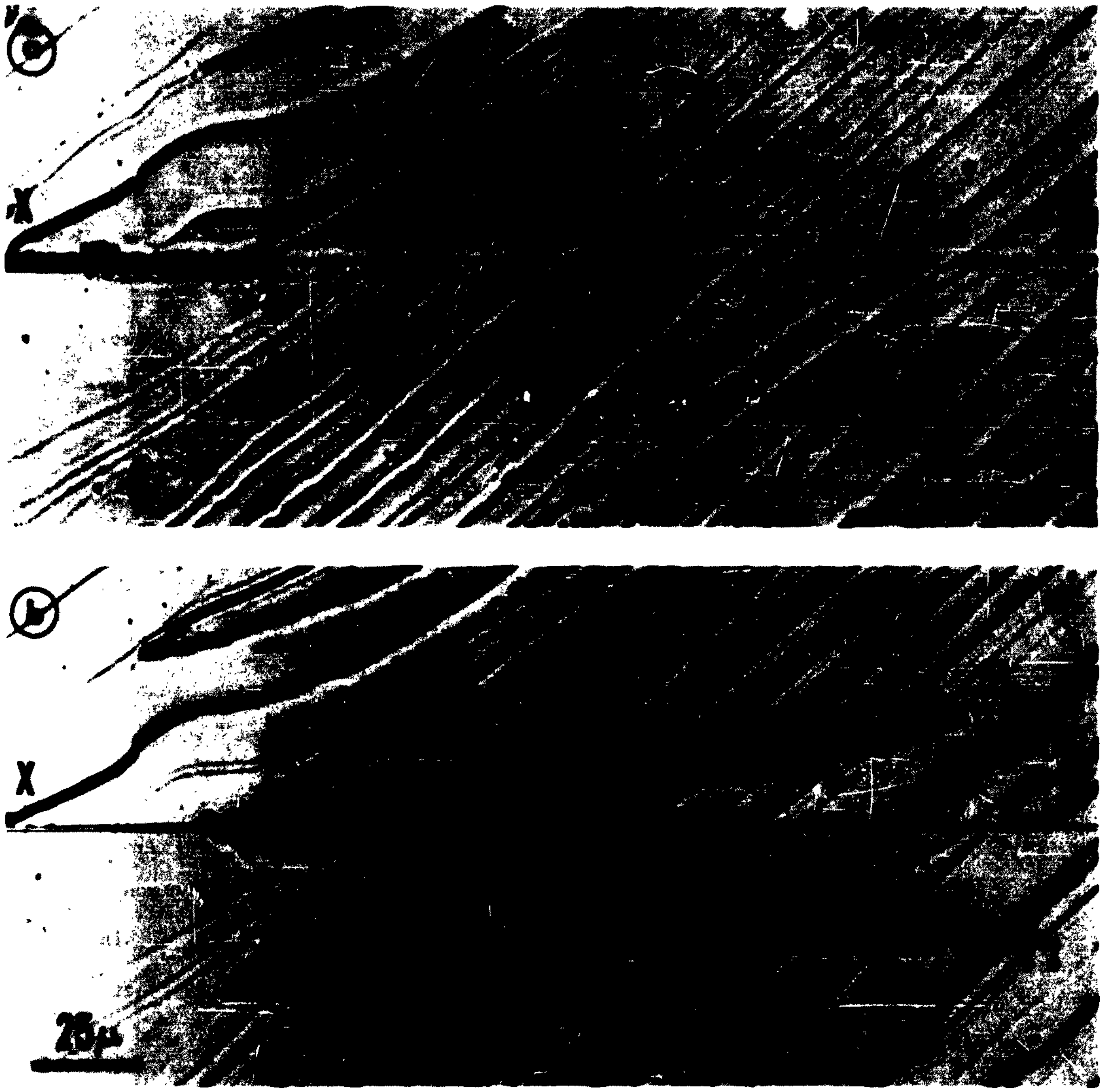

Figure 4. (a) Distribution of dislocations at tip (Y) of unpropagated cleavage crack. Dislocations between Y and Z probably {100} <100> type sessiles introduced by crack healing (crystal I, Face B, Fig. 3).

(b) Distribution of dislocations near original crack tip (Y) following repropagation. Note dislocation "wings". (crystal II, Face A, Fig. 3).

with the propagation of relatively long cracks. Such wings were only observed on the outer faces of crystals II (i.e. on faces A and A' in Fig. 3) when cracks of L_0 greater than about 2t were propagated. Examination of the surfaces EFGH, Fig. 3, produced by repropagation of the crack, revealed that such wings were associated with curve-fronted dislocation arrays of the type illustrated in Figs. 5(a) and 6(a). In the micrograph of Fig. 5(a), the crack front was originally at Y.

When short cracks ($L_0 < t$) were propagated, relatively narrow, straight-fronted dislocation arrays of the type illustrated in Fig. 5(b) were observed on the faces EFGH, and, in general, dislocation wings were not observed on the side faces A and A'.

A specimen exhibiting a propagation-induced dislocation array of the type shown in Fig. 5(a) was then cleaved as illustrated in Fig. 6(a) along the line K-L. Subsequent examination of the etched cross section JKLM demonstrated that dislocation wings are a free-surface induced phenomenon, as illustrated by Figs. 6(b), (c) and (d). Away from the sides A, A' of the crystal, for example at Y Figs. 5(a) and 6(a), dislocations associated with the propagation array extended into the crystal only $\sim 5\mu$. A further cross-sectioning of crystals II through QR, Fig. 6(a), confirmed that such propagation arrays as those illustrated in Fig. 5 merely consisted of a thin surface layer of dislocated material about 5μ deep. It seems likely that the dislocations constituting such arrays are {110} <110> type half-loops introduced while the propagating crack was accelerating from rest[16,17] and

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[16] J. J. Gilman, Trans. AIME, <u>209</u>, 449 (1957).

[17] A. J. Forty, Proc. Roy. Soc. (London), <u>A242</u>, 392 (1957).

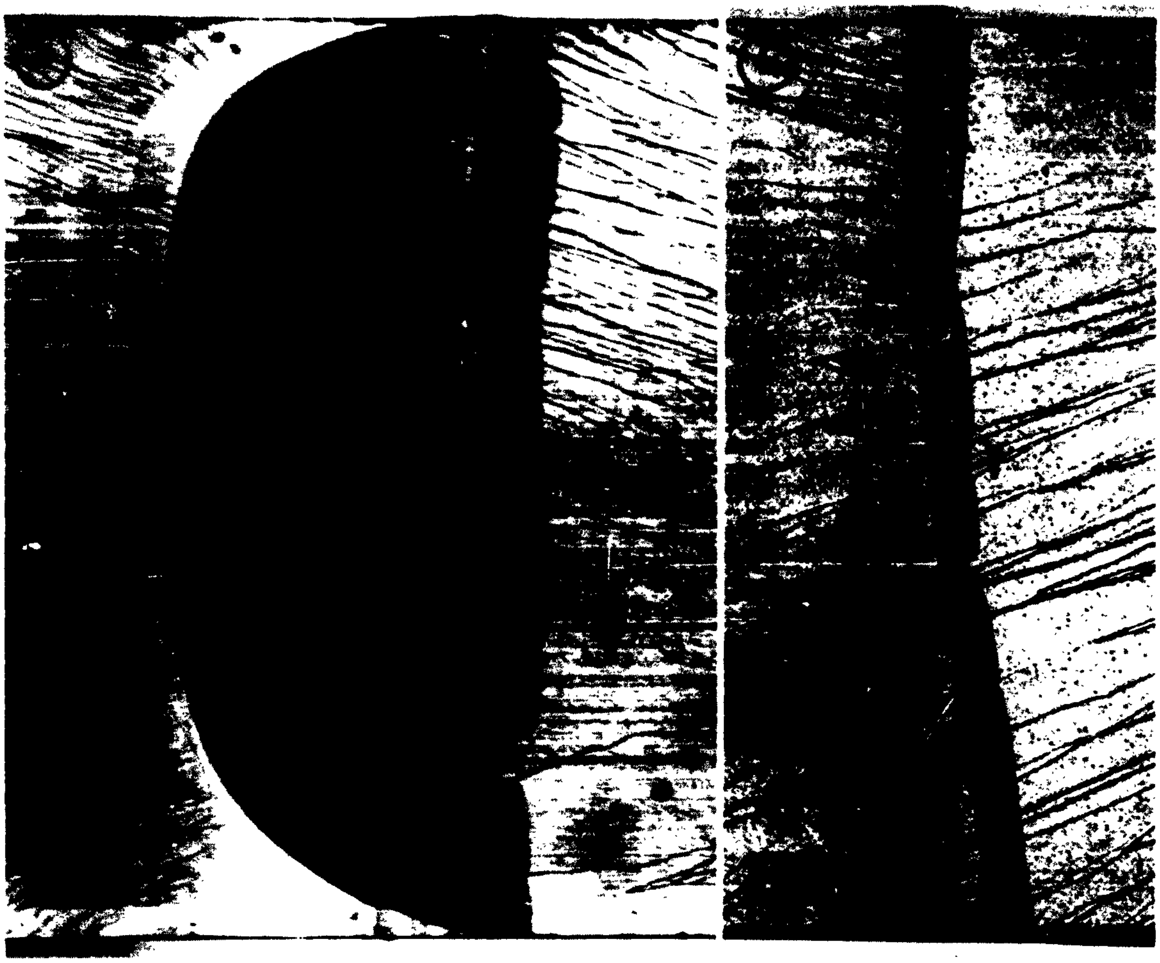

Figure 5. (a) Curve-fronted propagation array associated with cracks of $L_0 > 2t$. Original crack front at Y. (Face EFGH, Crystal II, Fig. 3).

(b) Narrow, straight-fronted propagation array associated with cracks of $L_0 < t$. Original crack front at Y (Face EFGH, Crystal II, Fig. 3).

before it reached its "critical" velocity[18] (above which dislocations are not generated).

It was stated earlier that tests utilizing relatively long cracks ($L_o > 2t$) often produce anomalously high values of the apparent surface energy. Furthermore, the metallographic studies just described have demonstrated that the propagation of such long cracks also introduces extensive surface arrays of dislocations - as first proposed by Orowan[19]. It would seem reasonable to suppose, therefore, that anomalously high values of γ_A and extensive surface deformation of the type illustrated in Fig. 5(a) might be related. Figures 7 and 8 demonstrate that this is indeed so. To obtain data for these figures, a representative group of the crystals used in the determination of γ_o, Fig. 2, was etched and examined. The extent of each propagation array, defined as x in Fig. 6(a), was determined and correlated with $(t/L_o)^2$ and γ_A. From Fig. 7 it can be seen that when $(t/L_o)^2$ is greater than about 0.25 ($L_o < 2t$), x remains relatively constant at about 0.05 cm. However, when $(t/L_o)^2$ is less than 0.25 ($L_o > 2t$), x is large and variable. Nevertheless, when $\gamma_A \geq \gamma_o$, the value of γ_A (quoted beside the points) varies in a consistent fashion with x, and the relationship between γ_A and x is shown in Fig. 8. The letters (a) and (b) in Figs. 7 and 8 refer to the specimens illustrated in Figs. 5(a) and 5(b) respectively.

SUMMARY

1) The surface energy of {100} MgO has been determined to be 1150 ± 80 ergs/cm^2 at 298°K.

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[18] J. J. Gilman, C. Knudsen and W. P. Walsh, J. Appl. Phys., 29, 601 (1958).

[19] E. Orowan, Rept's. Progress in Phys., 12, 185 (1948-49).

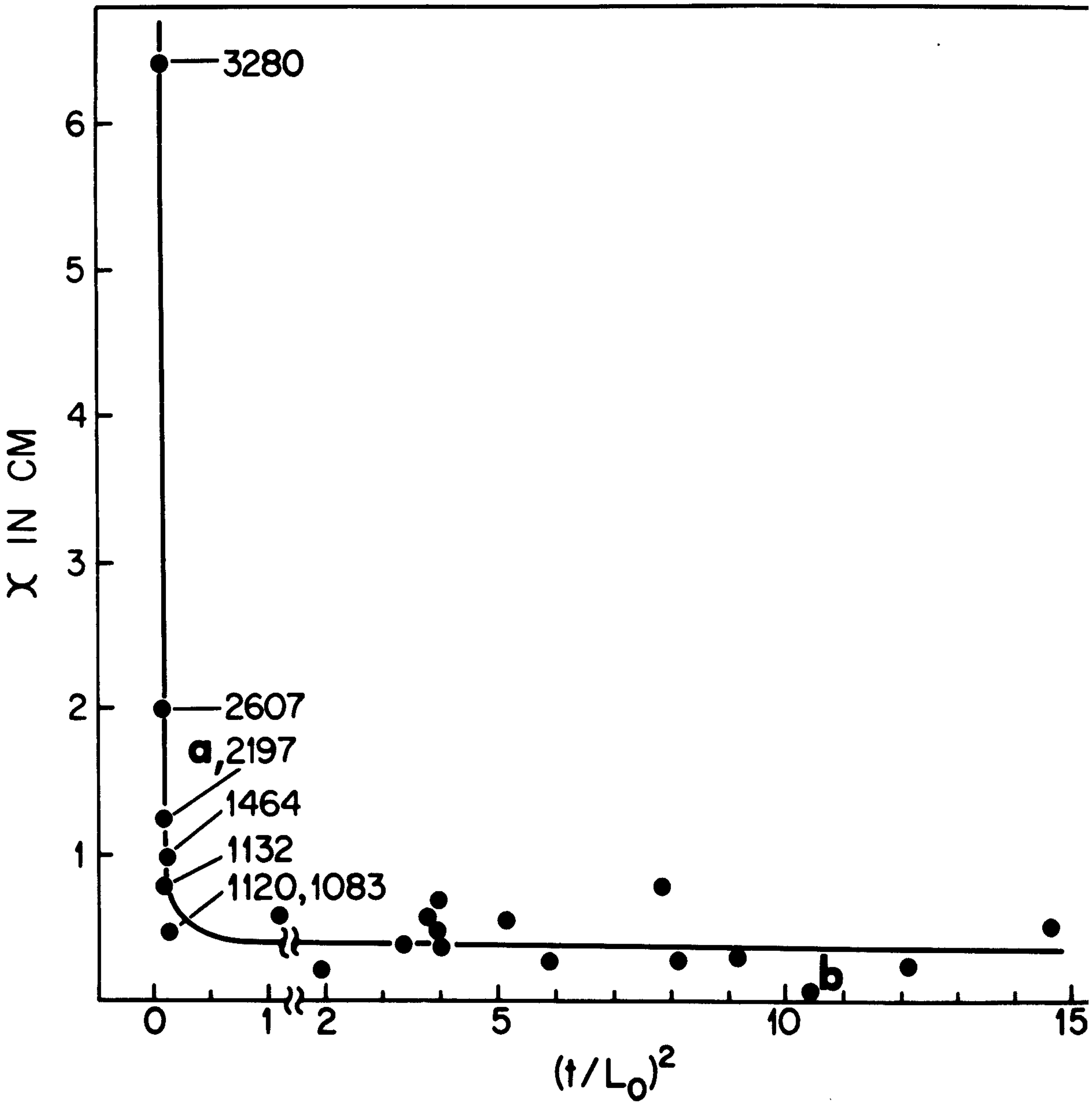

Figure 7. Relating metallographically observed extent of plastic deformation x (Fig. 6(with ratio $(t/L_0)^2$ and measured value of γ_A . Letters (a) and (b) refer to specimens illustrated in Figs. 5(a) and 5(b) respectively.

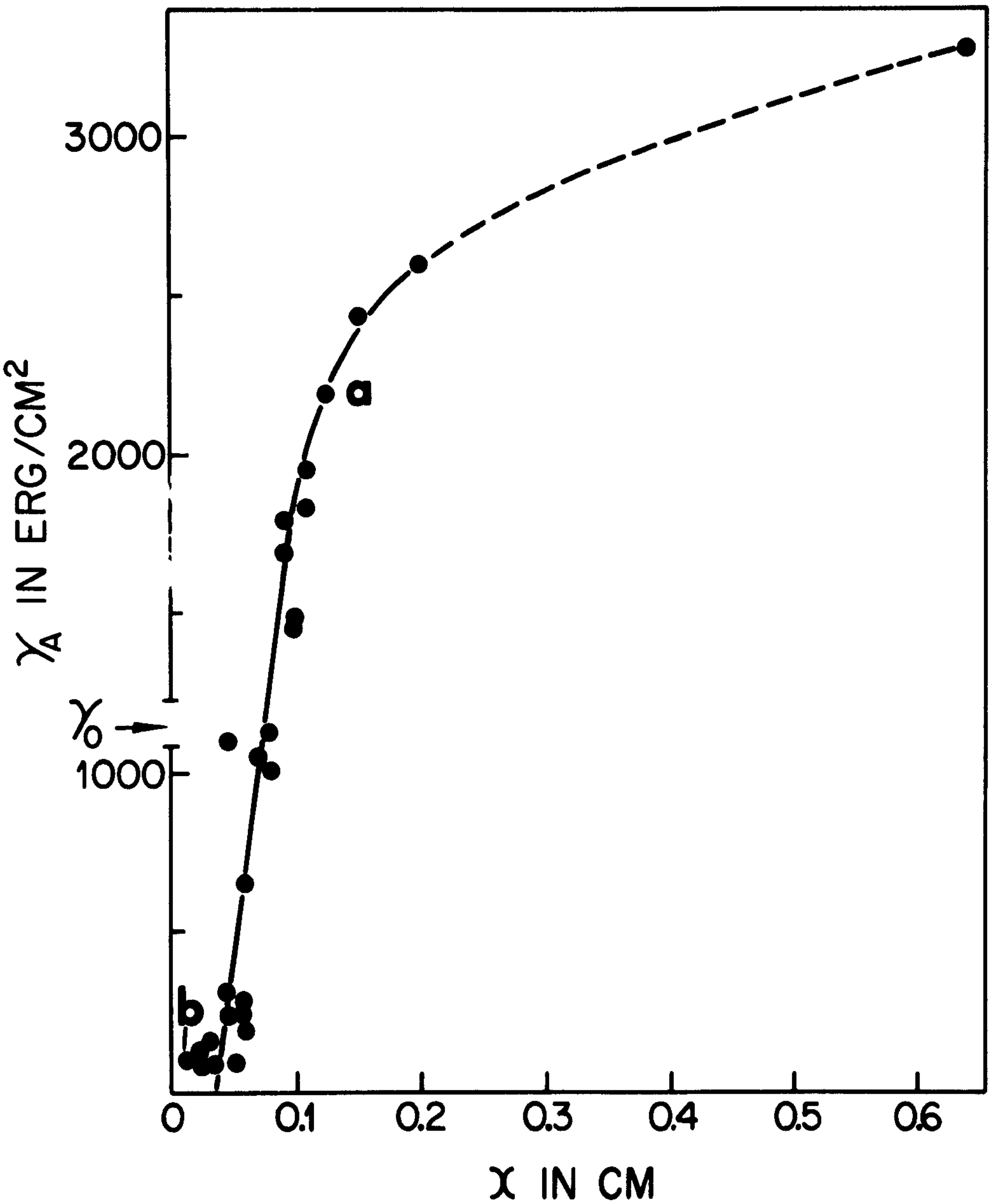

Figure 8. Illustrating relationship between measured value of γ_A and extent of plastic deformation x(Fig. 6(a)). Letters (a) and (b) refer to specimens illustrated in Figs. 5(a) and 5(b) respectively.

2) Metallographic studies have demonstrated that for cracks of $L_o > 2t$, the extent of plastic deformation in the vicinity of the crack tip and the measured value of the apparent cleavage surface energy are related.

ACKNOWLEDGMENTS

It is a pleasure to acknowledge the assistance of T. T. Hitch and H. Opperhauser in this work, which was supported in part by the Office of Naval Research under Contract Nonr-4162(00).

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